Kentucky Method 64-250-03
Revised 1/7/03
Supersedes 64-250-2000
Dated 5/8/2000

ANALYSIS FOR ISOCYANATE CONTENT

1. SCOPE: This method is designed for the determination of the isocyanate content of the resin system found in polyurethane paints. The reported isocyanate content is based upon 100% resin solids.

2. APPARATUS AND MATERIALS:

   2.1. Analytical balance

   2.2. High speed centrifuge

   2.3. Centrifuge tubes, minimum 50 mL heavy wall glass with vinyl lined screw caps

   2.4. 10 ml Syringes with Luer slip tip and caps

   2.5. 60 mm Aluminum weighing dishes

   2.6. Drying oven 110 ± 5°C

   2.7. Automatic titrator with stirrer (Mettler-Toledo DL 50 or equivalent)

   2.8. pH electrodes

   2.9. Fleaker or other vessel with stopper or cap, 200 mL minimum volume

   2.10. o-Xylene, reagent grade, dried over 3A Molecular Sieve (See 4.1)

   2.11. Buffer solutions, pH 4.00 and pH 7.00

   2.12. Dibutylamine, 99% assay

   2.13. Methanol, ACS reagent grade, dried over 3A Molecular Sieve (See 4.1)

   2.14. Hydrochloric acid solution, 1.0 N

3. PROCEDURE:

3.1. To analyze a one-component material, the resin system must first be separated from the whole paint.

3.2. Place a 40 ml sample of the whole paint into a high-speed centrifuge tube and cap immediately.

3.3. Centrifuge the sample to produce a minimum relative centrifugal force (RCF) of 4300 until the pigment and resin have separated. See 5.1 for RCF equation. See 4.2 for products requiring dilution to facilitate separation.

3.4. No separation is required for multi-component materials, when the resin system is packaged separately. The resin system is generally labeled “curing agent” or “part B”.

3.5. Determine the percent solids by weight of the resin solution (the resin solution is considered to be either the supernatant of a one-component material or the resin constituent of a multi-component material).

3.6. Condition the 60 mm aluminum weighing dishes in a drying oven at 110 ± 5°C for a minimum of 10 minutes and cool to ambient conditions in a desiccator.

3.7. Weigh 0.5 ± 0.1 g to 1.0 mg of the resin solution by difference from a syringe into a conditioned 60 mm aluminum weighing dish.

3.8. Immediately place the sample into a drying oven at 110 ± 5°C for 1.0 ± 0.25 hours.

3.9. Allow sample to cool to ambient conditions in a desiccator.

3.10. Weigh sample to 1.0 mg.

3.11. Calculate the percent solids by weight of the resin solution. See 5.2 for percent solids calculation.

3.12. Calibrate the titrimeter and pH electrodes with standard pH 7.00 and pH 4.00 solutions.

3.13. Place 25 ml of dry o-xylene into an appropriate size fleaker or vessel.

3.14. Weigh in the appropriate amount of resin solution. See 5.3 for sample weight equation.

3.15. Record the sample weight to the nearest 1.0 mg.

3.16. Add 20.00 ml of 1.7 N dibutylamine solution. Prepared by diluting 290 ml of dibutylamine to 1.0 L with dried o-xylene.

3.17. Cap the container immediately.

3.18. Allow the sample to stir for a minimum of 30 minutes but no longer than 60 minutes at room temperature.

3.19. Prepare a blank in the same manner, omitting the resin solution.

3.20. Wash down the sides of the containers with 100 mL of methanol.

3.21. Titrate the stirred blank with 1.0 N hydrochloric acid through the end point, which occurs at pH 5.0.

3.22. Record the volume of titrant used to the nearest 0.01 ml.

3.23. Titrate the stirred sample with 1.0 N hydrochloric acid through the end point, which occurs at pH 5.0.

3.24. Record the volume of titrant used to the nearest 0.01 mL.

3.25. Calculate the weight percent NCO. See 5.4 for weight percent NCO calculation.

4. NOTES:

4.1. Methanol and o-xylene used for this titration should be passed through separate columns containing 3A molecular sieve to remove any moisture that may be present. A recommended flow rate for passing methanol or o-xylene through a drying column is approximately 100 ml per minute. Dried stock may be collected into amber bottles and preserved by placing a small amount of 3A molecular sieve in the bottles.

4.2. Separation of pigment and resin of one-component materials may be aided by diluting the material up to the maximum volume of the centrifuge tube using dry o-xylene. Seal the centrifuge tube and shake the sample to obtain a homogenous sample prior to placing sample in the centrifuge.

5. CALCULATIONS:

5.1. Calculation for Relative Centrifugal Force (RCF):

$RCF = 0.00001118 \times r \times N^2$

Where, RCF = Relative Centrifugal Force
r = Rotating radius (cm)
N = Revolutions per minute

5.2. Calculation of weight percent resin solids of the resin solution:

$S_R = (W_2 / W_1) \times 100$

Where, S_R = Weight percent solids of the resin solution
W_2 = Weight of sample after drying
W_1 = Initial weight of sample

5.3. Calculation of the appropriate amount of resin sample to be used:

$W = (42 / NCO) / R$

Where, W = the weight in grams of the resin solution to be used
42 = the equivalent weight of an isocyanate group
NCO = the expected % NCO in the resin system
R = the percent weight solids of the resin solution

5.4. Calculation of the weight percent NCO of the sample:

$\%NCO = (((B-S) \times N \times 42) / (1000 \times (W \times R))) \times 100$

Where, B = the amount of titrant used to titrate the blank in milliliters
S = the amount of titrant used to titrate the sample in milliliters
N = the normality of the hydrochloric acid solution in equivalents per liter
42 = the equivalent weight of an NCO group
1000 = the factor converting liters to milliliters
W = the weight of the resin solution sample in grams
R = the percent weight solids of the resin solution

6. REPORT:

6.1. Report the percent weight solids of the resin solution to the nearest 0.01%.

6.2. Report the resin solution sample weight to the nearest milligram, 0.001 g.

6.3. Report the percent NCO of the resin system to the nearest 0.1%.

APPROVED _____
Director
DIVISION OF MATERIALS

DATE _____1/7/03_____

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